Research Article

Journal of Environmental Nanotechnology

Synthesis and Characterization of Fe Doped Manganese Oxide Nanoparticle by using Chemical Precipitation Method

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Received: 20.10.2020 Accepted: 14.12.2020

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ABSTRACT

Nanoparticles research is an area of intense scientific research due to a wide variety of potential applications in biomedical, optical and electronic fields. Manganese oxide nanoparticles can be utilized for advanced materials in batteries, water treatment and imaging contrast agents. In this present study, Fe doped Manganese Oxide nanoparticles were successfully synthesized by using the chemical precipitation method. The synthesized nanoparticles were characterized by using XRD, SEM, EDAX, FTIR, UV and PL. The average crystallite size of the sample had been investigated by the XRD technique. The morphology and grain size were obtained from SEM images. The elemental composition was confirmed by EDAX. The FTIR studies confirm the various functional groups present in the prepared sample. From the UV spectrum, the optical band gap was calculated. The intensity of emission radiation was absorbed by photoluminescence spectroscopy. This analysis confirmed that the Fe doped MnO has a slightly greater emission band than the pureMnO.

Keywords: Chemical precipitation; Ferric Chloride; MnO; PL.

1. INTRODUCTION

Nanotechnology is a branch of science, engineering and technology which deals with dimensions and tolerances of less than 100 nanometres of matter on an atomic or molecular scale. Nanoparticles have a wide application due to the unique size-dependent properties. Magnetic nanoparticles have been receiving considerable attention because of their wide range of applications, such as the immobilization of the proteins and enzymes, bio-separation, immunoassays, drug delivery, and biosensors. Nanoparticles possess a high surface to volume ratio due to their small size, which gives very distinctive features to nanoparticles (Sagadevan, 2015). The unique chemical and physical properties of nanoparticles make them extremely suitable for designing new and improved sensing devices, especially electrochemical sensors and biosensors. The important functions provided by nanoparticlesinclude the immobilization of biomolecules, the catalysis of electrochemical reactions, and the enhancement of electron transfer between electrode surfaces and proteins, labeling of biomolecules and even acting as reactant (Luo *et al.* 2006). Generally, metal oxide nanoparticles are inorganic. Various nanoparticles like Fe, Ni, Co, Mn, and Zn are enormously accepted magnetic material for a wide range of applications like magnetic sensors, recording equipments, telecommunications, magnetic fluids, microwave absorbers etc. Among various metal oxide nanoparticles, manganese dioxide is an important transition metal oxide of P-type semiconducting materials. Generally, nanoparticles have been prepared by physical vapour deposition, chemical vapour deposition, aerosol processing, sol-gel process, reverse micelle method, mechanical milling etc. A wet chemical technique such as hydrothermal sol-gel, emulsion and conventional co-precipitation method is commercially widely used because of its cost-effective nature (Cherian *et al.* 2016). Manganese oxides (MnO_2) are natural components of soils, aquifers and sediments, and are known to be strong adsorbents of metal ions. They are important materials with a variety of applications in different fields such as chemical sensing devices, magnetic devices, environmental pollution absorbent, catalysis, ion-sieves, rechargeable batteries, hydrogen storage media and microelectronics (Dang *et al.* 2015).

2. EXPERIMENTAL DETAILS

2.1 Chemicals and Reagents

Manganese chloride, NaOH and Ferric chloride were used. Distilled water was used as a solvent throughout the experiment.

2.2 Synthesis of MnO nanoparticles

Take 4g of manganese chloride, and it was dissolved in 50 ml distilled water and stirred for 30 minutes using a magnetic stirrer. Then take 3g of NaOH pellets were dissolved in 20 ml distilled water was added

to the solution to maintain the pH level at 8. After 5 hours of stirring, the brown colour precipitate was obtained and filtered by filter paper. The collected precipitate was kept in a microwave oven at 75W for 10 minutes. Then it was dried in a muffle furnace at 400 °C for 4 hours. The dried nanoparticles were taken in a mortar and make into a powder.

2.3 Synthesis of Fe doped MnO nanoparticles

Take 4g of manganese chloride, and it was dissolved in 50 ml distilled water and stirred for 30 minutes using a magnetic stirrer. Then take 3g of NaOH pellets were dissolved in 20 ml distilled water was added to the solution to maintain the pH level at 8. 1g of Ferric chloride dissolved in 10ml of distilled water was added to the above solution. After 5 hours of stirring, the dark brown colour precipitate was obtained and filtered by filter paper. The collected precipitate was kept in a microwave oven at 75W for 10 minutes. Then it was dried in a muffle furnace at 400 °C for 4 hours. The dried Fe doped nanoparticles were taken in a mortar and make into a powder.

3. RESULTS & DISCUSSION

3.1 XRD Analysis

The XRD pattern for Fe doped MnO nanoparticles was shown in Fig. 1. The narrow peaks in the XRD pattern indicate the crystalline nature of nanoparticles and the peaks corresponding to 2θ values = 36.2°, 56.1°, 58.2°, 62.4° which indicates the miller indices (101), (110), (220), (103) respectively and represent the hexagonal shape. These results were good compared to the standard data of JCPDS card no: 41-1442. The crystallite size of the Fe doped MnO nanoparticles was found by this XRD analysis is 11.5 nm. The crystallite size of the particles was calculated by using the Debye-Scherrer formula,

$$D=k\,\lambda/\beta\cos\theta$$

where, D - Crystallite size, λ - Wavelength of the X-Ray source (1.5406 Å), β - Full width at half maximum of the diffraction peak, k - Scherrer constant with value 0.9 to 1 and θ - Bragg's angle.

Table 1. XRD result of Fe doped MnO nanoparticles

Sample	2θ	FWHM	Crystallite Size (nm)	Average Size (nm)
Fe doped MnO	36.2	0.78980	10.4938	11.5706
	56.1	0.52070	16.8742	
	58.2	0.77110	11.4855	
	62.4	1.21250	7.42887	

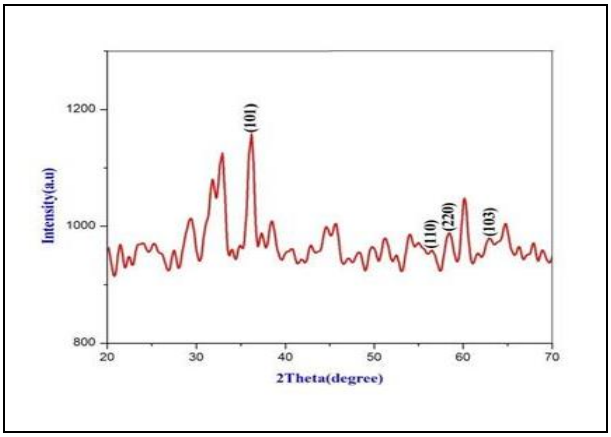

Fig. 1: XRD pattern for Fe doped MnO nanoparticles

3.2 SEM Analysis

Scanning Electron Microscope was used to deduce the particle size and morphology of the synthesized Fe doped manganese oxide nanoparticles. Fig.2 shows that the particles were nearly sphere-like structure. They clearly show randomly distributed grains with smaller size. The size of the synthesized Fe doped manganese oxide nanoparticles was found to be in the range of 40-71.44 nm.

3.3 EDAX Analysis

EDAX analysis is used to indicate the elemental composition present in the sample. Manganese (Mn), Oxide (O) and Iron (Fe) peaks were observed in fig. 3 indicating the presence of Fe doped MnO nanoparticles.

3.4 FT-IR Analysis

Fourier Transform Infrared Spectroscopy (FTIR) identifies chemical bonds in a molecule by producing an infrared absorption spectrum. It was used to analyze the functional groups, and other impurities present in the Fe doped Manganese Oxide nanoparticles are shown in fig. 4. FT-IR spectra of Fe doped Manganese Oxide are recorded in the range of 400-4000 cm^{-1}. The absorption spectrum in the excitation of bond gives the various stretching of functional groups.

3.5 UV Visible Spectroscopy Analysis

The optical property and bandgap energy of the samples were determined from UV absorbance. The UV visible spectra of Fe doped MnO nanoparticles were shown in Fig.5. The spectra are recorded in the range of 200-900nm. The maximum absorbance peak was at 223 nm. The bandgap energy (Eg) value of the Fe doped MnO nanoparticle was 5.57 eV. The bandgap energy was calculated by,

$$Eg = h\upsilon = hc / \lambda$$

Where,

h = Planck's constant (6.626×10^{-34} m^2kg/s),
λ = Wavelength (nm),υ - Frequency, c - Speed of the light (3×10^8 m/s)

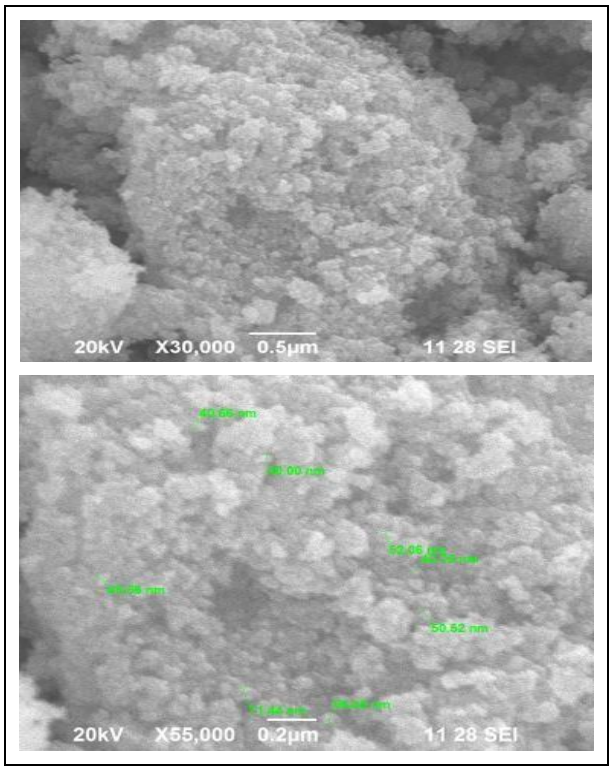

Fig. 2: SEM images of Fe doped MnO nanoparticles

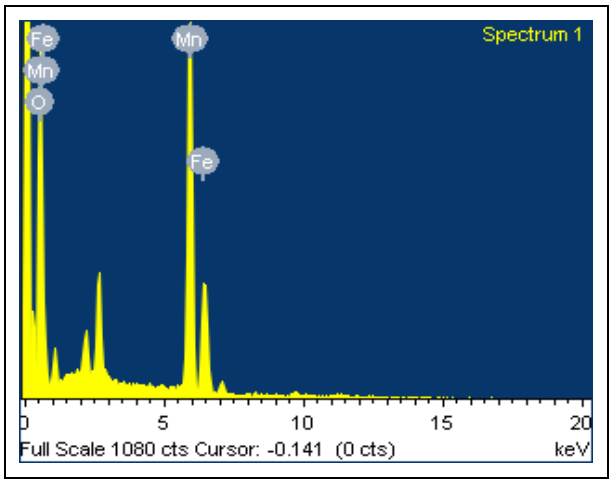

Fig. 3: EDAX analysis for Fe doped MnO nanoparticles

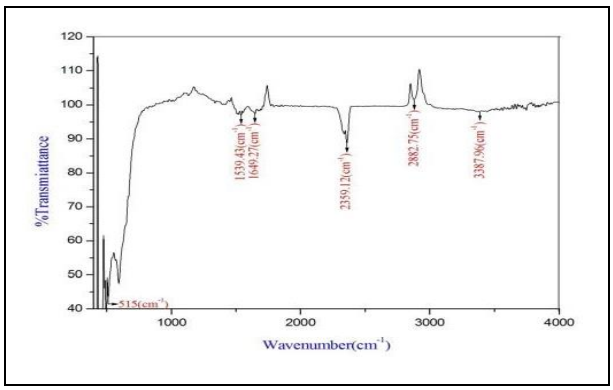

Fig. 4: FTIR spectrum of Fe doped MnO nanoparticles

Table 2: FTIR peaks of Fe doped MnO nanoparticles

Band Range (cm^{-1})	Stretching	Intensity
1539.43	N=O	Strong
1649.27	C=O	Strong
2359.12	NH+	Strong
2882.75	Symmetric CH	Very Strong
3387.96	O-H	Strong
515	Mn-O	Strong

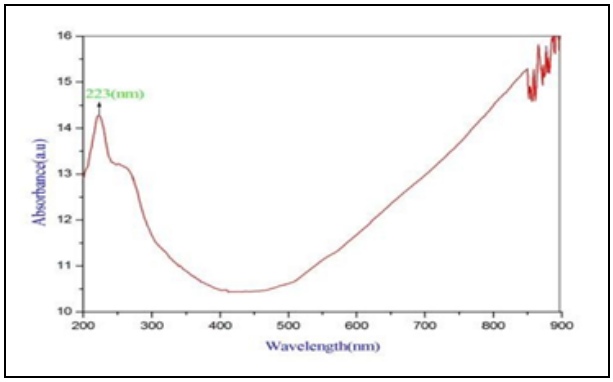

Fig. 5: Absorption spectra of Fe doped MnO nanoparticles

3.6 Photo Luminescence Analysis

The intensity of emission radiations was absorbed by photoluminescence spectroscopy. The emission radiation of Fe doped MnO nanoparticles were shown in figure 3.6. The excitation wavelength occurs at the range of 269.8nm for pure and Fe doped MnO nanoparticles. The emission band of pure and Fe doped MnO nanoparticles were located at 295nm and 296 nm.

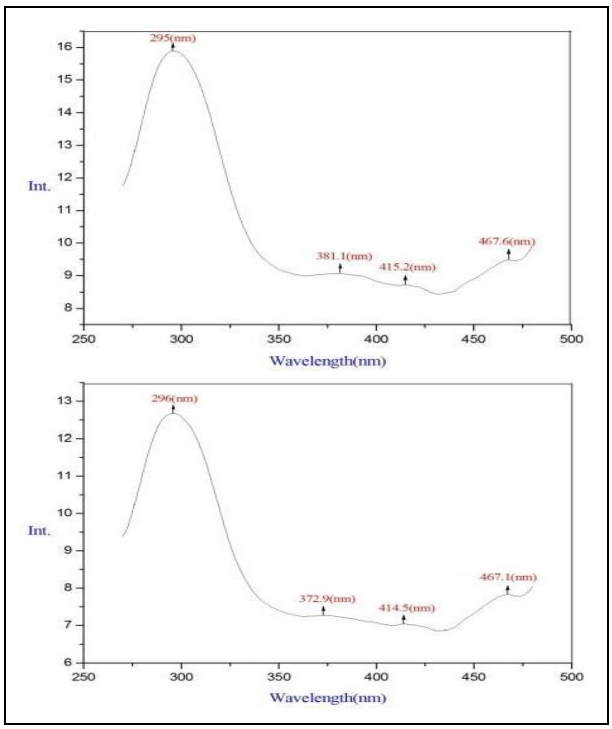

Fig. 6: (a) Photoluminescence Analysis of pure and Fe doped MnO nanoparticles.

4. CONCLUSION

In this present study, the pure and Fe doped MnO nanoparticles had been synthesized by using the chemical precipitation method. It had been characterized by using XRD, SEM, EDAX, FTIR, UV and PL, respectively. The crystallite size of Fe doped MnO nanoparticles prepared by chemical precipitation method is 11.5nm, and it had a hexagonal shape. SEM images showed a nearly sphere-shaped morphological structure. The presence of elemental composition was confirmed by EDAX. The FTIR studies confirm the various functional groups present in the prepared sample. In UV analysis, the bandgap energy for Fe doped MnO nanoparticle was at the range of 5.57nm. The emission band of pure and Fe doped MnO nanoparticles were located at 295nm, and 296 nm was analyzed by photoluminescence. The Fe doped MnO nanoparticle had a slightly greater emission band than the pure MnO nanoparticle.

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